

# Metastable states of a flux line lattice studied by transport and Small Angle Neutron Scattering

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Flux Lines Lattice (FLL) states have been studied using transport measurements and Small Angle Neutron Scattering in low  $T_c$  materials. In Pb-In, the bulk dislocations in the FLL do not influence the transport properties. In Fe doped  $NbSe_2$ , transport properties can differ after a Field Cooling (FC) or a Zero Field Cooling (ZFC) procedure, as previously reported. The ZFC FLL is found ordered with narrow Bragg Peaks and is linked to a linear  $V(I)$  curve and to a superficial critical current. The FC FLL pattern exhibits two Bragg peaks and the corresponding  $V(I)$  curve shows a S-shape. This can be explained by the coexistence of two ordered FLL slightly tilted from the applied field direction by different superficial currents. These currents are wiped out when the transport current is increased.

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Flux Line Lattice (FLL) order and its relationship with the pinning and dynamical properties provides an excellent model system [1]. The competition between the FLL elastic properties and the quenched or thermally driven disorder can lead to different vortex matter states. To some extent, this implies also changes in the transport properties. A good example is the peak effect observed in several type II superconductors, a sudden increase of the critical current close to the superconducting-normal transition, which has been for a long time considered as a proof of a disordering transition. The basic idea was from Pippard [2] who noted that the pinning threshold for dilute bulk pinning goes quadratically to zero near the second critical field. Since this is faster than the elastic interaction between vortices, bulk pinning centers can become more effective on a less rigid lattice at high field and that can lead to a peak effect. Larkin-Ovchinnikov collective pinning model [3] and the associated scaling arguments gave a more precise theoretical foundation to the link that is usually made between the loss of FLL order and the high critical current. It follows that a high critical current is *a priori* associated to a disordered FLL. Nevertheless, these arguments suggest that the peak effect should occur more often than it is actually found. Numerous experiments have also shown that the link between the FLL order and the critical current is far from being direct, in contradiction with the previous assumptions. Thorel's neutron scattering pioneering experiments have first shown that the Flux Lines Lattice (FLL) quality can be modified without chang-

ing the critical current [4]. One possible explanation is that the relevant length scale associated with the critical current is not the length scale of topological order [5]. Another possibility, already proposed by different authors [6, 7, 8], is that the order of the FLL in the bulk is not the most important parameter for understanding the transport properties of the FLL.

In addition to the peak effect, very peculiar transport properties can be observed. In particular, whereas Voltage(Current) ( $V(I)$ ) characteristics are usually reversible and do not depend on the way from which the FLL is prepared, hysteretic  $V(I)$  curves are observed in  $NbSe_2$ , when the FLL is formed after a Field Cooling (FC) [9, 10]. A model has recently emerged, and is supported by different experiments [11]. The key ingredients of this model are a supercooling of a high temperature/high critical current state into a low temperature/low critical current state, and an annealing effect over surface barrier. In order to explain the high critical current phase, a strongly disordered FLL is involved. This amorphous or liquid-like state is in favor of a genuine phase transition when crossing the peak effect. Nevertheless, very little is known about the real structure of these phases. There is even contradictory and puzzling results. Indeed, recent decoration experiments have shown that no disordering transition can be evidence in the peak effect region of pure or Fe doped  $NbSe_2$  samples [12]. The high critical current FLL state remains unexplained. Nevertheless, it should be specified that such experiments give the position of field lines only when they protrude from the sample surface. It can not be excluded that this distribution differs in the bulk.

Small Angle Neutron Scattering appears as a complementary technique, since the order of the FLL can be

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investigated *in the bulk* of the material. It is also possible to measure in real time  $V(I)$  curves together with the FLL diffraction patterns and hence to investigate the relationship between the transport current properties and the FLL structure. The aim of the following experiments is to use SANS in order to compare different FLL states with respect to their dynamical properties. After discussing a more conventional case, we will focus on the FLL states close to the peak effect in  $\text{NbSe}_2$ .

The SANS experiments were performed in the Laboratoire Leon Brillouin (Saclay, France). Large single crystals of Fe doped  $2\text{H-NbSe}_2$  (200 ppm of Fe, size  $8 \times 6 \times 0.5 \text{ mm}^3$ ,  $T_c = 5.5 \pm 0.15 \text{ K}$  measured by specific heat) and of polycrystalline Pb-In (10.5 % of In by weight, size  $30 \times 5.5 \times 0.5 \text{ mm}^3$ ,  $T_c = 7.05 \pm 0.05 \text{ K}$ ) were used. The magnetic field was applied parallel to both the  $c$ -axis of the crystal and the incident neutron beam. The scattered neutrons ( $\lambda_n = 10 \text{ \AA}$ ,  $\Delta\lambda_n / \lambda_n \approx 10\%$ ) were detected by a 2D detector located at a distance of 6.875 m. In the following,  $\omega$  will refer to a rotation around the vertical axis, and  $\phi$  to a rotation around the horizontal axis (Fig. 1). Superconducting leads were attached using Indium solder pressed between copper slabs. At the working temperature of 2 K (in superfluid liquid He), this allows an injection of about 10 A without any overheating. We present here results obtained for a magnetic field of 0.4 T and 0.2 T.

Before describing the results obtained in Fe doped  $2\text{H-NbSe}_2$ , it can be helpful to present a more conventional case, where no peak effect and no hysteretic  $V(I)$  curves are observed. In Pb-In samples, the dynamical properties of the FLL are well documented. For a state of the FLL phase diagram (B and T fixed), only one critical current is measured. The  $V(I)$  curves are found reversible when they are cycled. It does not necessary mean that the FLL is always the same and that no metastable states are present, but it rather means that the structure of these possible states does not play a significant role for pinning and transport properties. For example, we have reproduced an experiment described in [13], but focusing on the details of the  $V(I)$  curves. The goal is to follow the evolution of a moving FLL in a polycrystal of Pb-In (Fig. 2). The 2D pictures consist in 12 acquisitions taken at a particular  $(\omega_i, \phi_i)$  angular position, in order to fulfill at the best the Bragg conditions. Without any applied current, we observe a powder-like diffraction pattern meaning that the FLL is highly dislocated and that its orientational order is strongly degraded. This is due to the interaction between the randomly oriented crystal axis of the sample and the FLL unit cells. The FLL is ordered within the grains of the Pb-In slab but FLL dislocations are present in order to accommodate the shear strains. The size of the crystal grains is optically evaluated to be about  $100 \mu\text{m}^2$ , which indeed corresponds to large ordered domains (thousands of vortices). On the other hand, at the scale of the sample, this corresponds also to thousands of ordered domains. This is enough to observe a ring of scattering due to the poor azimuthal

resolution of this technique. When increasing the current above the critical current, the FLL order becomes strongly improved. The dislocations are expelled when the flux lines are moving in the sample and long range orientational order is established. The corresponding ordered state can be frozen (experimentally, we turned off the current during the FLL flow). On the contrary, dislocations are appearing again when the current is slowly decreased, which means that they can be considered as equilibrium features. It is thus possible to stabilize a FLL with crystalline order or a FLL with many dislocations and to analyze the  $V(I)$  curve corresponding of each FLL state. We measured in both cases the same critical current and even exactly the same  $V(I)$  curve (Fig. 2). These results show that the presence of large scale bulk dislocations in the FLL governs orientational order but may have no effect on the critical current or on the main dynamical properties. One should also refer to Thorel *et al* [4] who observed different qualities of the vortex crystal that do not affect the critical current, even in monocrystalline slabs of Niobium. In summary of this part, large scale bulk dislocations are found to have no link with the critical current and with the dynamical properties of the FLL. They can not be *a priori* involved to interpret a high critical current state of the FLL.

We performed the same kind of experiments in crystals of  $\text{NbSe}_2$ , in order to compare the different states of the FLL which could be responsible for the anomalous transport properties. We have first observed the simplest case: the FLL after a Zero Field Cooling (ZFC). Fig. 3a shows the SANS pattern we have obtained. This corresponds to an ordered hexagonal FLL. The scattering wave vector  $Q = 0.00953 \pm 0.00050 \text{ \AA}^{-1}$  is in good agreement with the theoretical one,  $Q_0 = 0.00938 \text{ \AA}^{-1}$  calculated for the regular crystal of flux lines. The alignment of the FLL is along both the  $a$ -axis and the lateral faces of the crystal. No difference is observed on the 2D pictures for different FLL velocities  $V_L$  ( $V_L = E/B$ , with  $E$  the electric field and  $B$  the magnetic field). The orientational order is thus preserved, confirming that, as it was observed in other type II superconductors [14], the FLL is well ordered and moves as a whole during the flux flow. We also performed  $\omega$  rocking curves. Small widths are obtained by analyzing the peaks with Lorentzian fits (Fig. 3b). We obtain  $\Delta\omega$  ( $FWHM$ ) =  $0.232 \pm 0.020 \text{ deg}$  for the FLL after ZFC and without external current applied. This is close to the experimental resolution given by the angular divergence of the beam (0.150 deg). If we increase the transport current, but staying below the critical current value, absolutely no change is observed. When the applied current is higher than the critical current  $I_c = 2.5 \text{ A}$ , a slight increase of the RC width  $\Delta\omega$  is observed. The reason is that the transport current  $I$  imposed by the external generator has to fulfill the Maxwell-Ampere equation. As the moving Bragg planes are observed to be strictly translationally-invariant [14], one can neglect the in-plane field gradient and the Maxwell-Ampere equation reduces to  $\mu_0 J_y = \partial B_x / \partial z$  which physically represents

a curvature of the field lines over the thickness of the sample. This bending is responsible for the increase of the rocking curve width [15]. This gives a direct measurement of the amount of transport current flowing *in the bulk* of the sample, via a simple integration of the Maxwell-Ampere equation ( $I_{bulk} \approx \frac{2WB}{\mu_o \Delta \omega}$ ). Bulk and surface currents can thus be distinguished (see [16] for details). The corresponding variation of  $I_{bulk}$  as function of the applied current  $I$  is shown in the fig. 4. It is clear that the large error bars due to the experimental resolution (given mainly by the mechanical precision during the sample rotation) combined to the relatively small number of experimental points do not allow to determine precisely the distribution of the current. It is nevertheless quite reasonable to estimate that no bulk current is present for  $I < I_c$  and that a bulk current, roughly  $(I - I_c)$ , is observed for  $I > I_c$ . The observed linear  $V(I)$  curve [16] and the critical current values and variations [17] are complementary indications in favor of a surface pinning mechanism in NbSe<sub>2</sub>.

Concerning SANS measurements coupled with transport experiments in NbSe<sub>2</sub>, let us compare with Yaron *et al* experiments [18], which purpose was to measure the longitudinal correlation length characteristic of FLL order. Yaron *et al* observed a narrowing of the rocking curve when  $I > I_c$ . They attributed this effect to an improvement in the FLL order. On the contrary, we observe here, what was previously observed in other superconductors [15, 16], that the rocking curve broadens when the over critical current penetrates the bulk. As this is a simple consequence of the Maxwell equations, it appears not clear to us why such effect was not observed by Yaron *et al*. A possible interpretation is that the reported Rocking curves are performed in the direction perpendicular to the one reported in the present experiment. In such case and as observed in Nb-Ta samples [13], a very small narrowing of the rocking curve can be observed. It can reasonably be attributed to an enhancement of the homogeneity of the FLL Bragg planes spacing (due to the homogeneous bending in the perpendicular direction) rather than to a change in a correlation length. In any case, if this length can have a clear definition in static, it has to be taken with care for *curved* moving flux lines.

The FLL in NbSe<sub>2</sub>, formed after a ZFC, appears to be quite similar to the FLL in conventional type II superconductor with a moderate critical current. More differences are expected after a FC, because in this case the  $V(I)$  curve exhibits a very peculiar shape. The samples we used for the SANS experiments are larger than those usually employed for transport properties and we have to precise that we have measured  $V(I)$  curves (Fig.5c) very similar to what was already studied in details by others [11, 19, 20]. In very short, they are hysteretic, with a S shape for the first ramp of current after the FC and a linear shape and a reversible behavior for all the following ramps.

Obtaining information on the FLL structure prepared after FC was not immediate. For the same centering as

for the ZFC case, any scattered intensity can be observed on the 2D multi-detector. The first thought was that the FLL was so strongly disordered that the Bragg peaks were considerably broadened. But this is not the right reason, as evidenced in Fig.5a where the corresponding  $\omega$  rocking curve is shown. Compared with Fig. 3b, one can realize that the Bragg condition has changed and above all that the rocking curve exhibits a double peak, what is far from being expected. The sum of the integrated intensity contained in these two peaks fits, within error bars, to the integrated intensity of the Bragg peak of the FLL after ZFC, and the widths of the peaks are comparable too. Consequently, we can not explain these strange Bragg peaks involving a FLL disorder in its proper sense. More likely, they should correspond to two very similar FLL which are ordered, but slightly tilted from the applied magnetic field direction from few tens of degrees. An interpretation in terms of a rotation due to a Doppler shift can be eliminated because the FLL frame is not moving. A more reasonable possibility is that we are observing two FLL possessing two different Bragg planes spacing because of different magnetic densities. This assumption would imply in the sample a magnetic field gradient of more than half ( $\Delta B \approx 0.2T$ ) the applied one. This looks hardly compatible with the strong interaction between the flux lines which limits the compressibility of the vortex array. Furthermore, as evidenced in the figure 6, no change is observed on the length of the Q vector, meaning that the average magnetic field density inside the sample does not suffer from such strong heterogeneity. Finally, we propose that these two peaks are a signature of the "two phases" observed by Marchevski *et al* using scanning hall ac probe [21]. Their experiments evidenced that two states possessing different critical currents are coexisting in the region of the peak effect. Our SANS experiment offers complementary information. The fact that the two peaks are very similar is not in favor of two states with a different bulk underlying disorder. The shift between these two peaks indicates that the two FLL are slightly tilted by static and small in plane field components. Let us call  $+b_1$  and  $-b_2$  these components. The center of the Bragg peaks are turned by 0.13 and -0.40 deg from the initial Bragg condition. It follows that  $b_1 = 4000 \tan(0.13) \approx 9$  G and  $-b_2 = 4000 \tan(-0.35) \approx -24$  G. These field components should be induced by a peculiar and non symmetric distribution of superficial currents. Following previous authors [19], we adopt the point of view that the edge of the sample is the region of the highest currents. Both Bragg peaks cover roughly the same surface. We can thus estimate that the width of the sample is divided into two parts of the same size, i.e. 3 mm for each. We know that the low critical current is 2.5 A and that it corresponds to a superficial value of  $i_{low} = I_c/2W \approx 2A/cm$ . At the same time, we have measured, when the peak effect is at its maximum, a ratio  $\frac{I_{high}}{I_{low}} \approx 7$ . With the reasonable assumption that it corresponds to a state where the high critical current state invades all of the sample, we can deduced that

$i_{high} \approx 7i_{low} \approx 14 A/cm$ . Using the Ampere theorem and after a superposition with the top and bottom surfaces, we find that the two domains transporting  $i_{high}$  and  $i_{low}$  generates bulk components of magnetic field which are  $b = \mu_0 i \approx 2.5$  and 18 G (Fig. 7). This is not so far from the measured values (9 and 24 G), considering this highly schematic picture.

In this picture, the FC state is characterized by large loops of current, that are as many non dissipative paths for a transport current. Increasing the transport current induces a preferential direction and one loop should be turned off. This implies that one of the two tilted FFL has to disappear. This is indeed observed in fig.5b. If the transport current is increased again up to the high critical current, the second loop disappear. All the flux lines are now along the main magnetic field direction and the Bragg angle returns close to its normal value (Fig.5c). Finally, the surfaces can not transport anymore non-dissipative current, the excess penetrates the bulk and the current flow becomes resistive. The  $V(I)$  curve returns to a classical behavior in the linear form  $V = R_{ff}(I - I_c)$ ,  $I_c$  being the low critical current. Since the initial loops have been cleaned by the current, they have no reason to reappear and the  $V(I)$  curve is then observed reversible. We note that this annealing-like effect by subcritical superficial current is in good agreement with the magneto-optical results observed in the reference [11].

One of the remaining (and central) question is why FC is responsible for such a non usual state, whereas it is classically the procedure used to obtain a FLL close to its equilibrium state. First, this is correlated to the presence of the peak effect in the critical current. This latter can be understood as superimposed on the "normal" critical current [17] [23]. If the sample is doped (here with Fe) or if impurities are present, the peak effect generally broadens and the metastability observed in transport measurements after FC become very obvious. This metastability is likely due to the (dynamical) coexistence of large regions possessing two critical currents [11], whom origin remains unknown. We propose that these currents are in fact superficial. This has to be put

close to old results obtained in conventional type II superconductors: A thin surface film of copper was shown to suppress the peak effect in Pb-Tl ribbons [23], what evidences a mechanism governed by surface currents. For a sample doped with impurities, small broadening or even small differences between the critical fields, bulk  $B_{c2}$  or surface  $B_{c3}$ , can reasonably be expected. We can speculate that this is a reason for a heterogeneous FC and for the corresponding freezing of metastable surface currents. Looking at the influence of surface treatments such as metal plating in  $NbSe_2$  appears thus to be particularly interesting in order to confirm the role of the surface currents.

In summary, we have studied by SANS the structure of the flux lines lattice and its link with dynamical properties. In Pb-In, a conventional type II superconductor,  $V(I)$  curves are the same whatever the FLL state. On the contrary, a very peculiar behavior is observed in Fe doped  $NbSe_2$  for the FC case. The diffraction pattern exhibits two FLL, shifted by tenth of degrees, corresponding to about tens of Gauss perpendicular to the applied magnetic field. These extra-field components require the presence of two loops of quasi-equilibrium currents possessing different values. This leads to an annealing mechanism by sub critical currents confirming previous results obtained by ac scanning probe [11]. Nevertheless, the FLL with the high critical current appears not to be a bulk disordered state, like an amorphous or a glassy state with a large amount of bulk free dislocations, but rather to be a state similar to the ordered FLL with the moderate critical current. This can be explained by the superficial nature of the critical currents. Of course, all questions concerning the physical origin of this higher and unstable current remain open and more experiments are needed to support this proposition.

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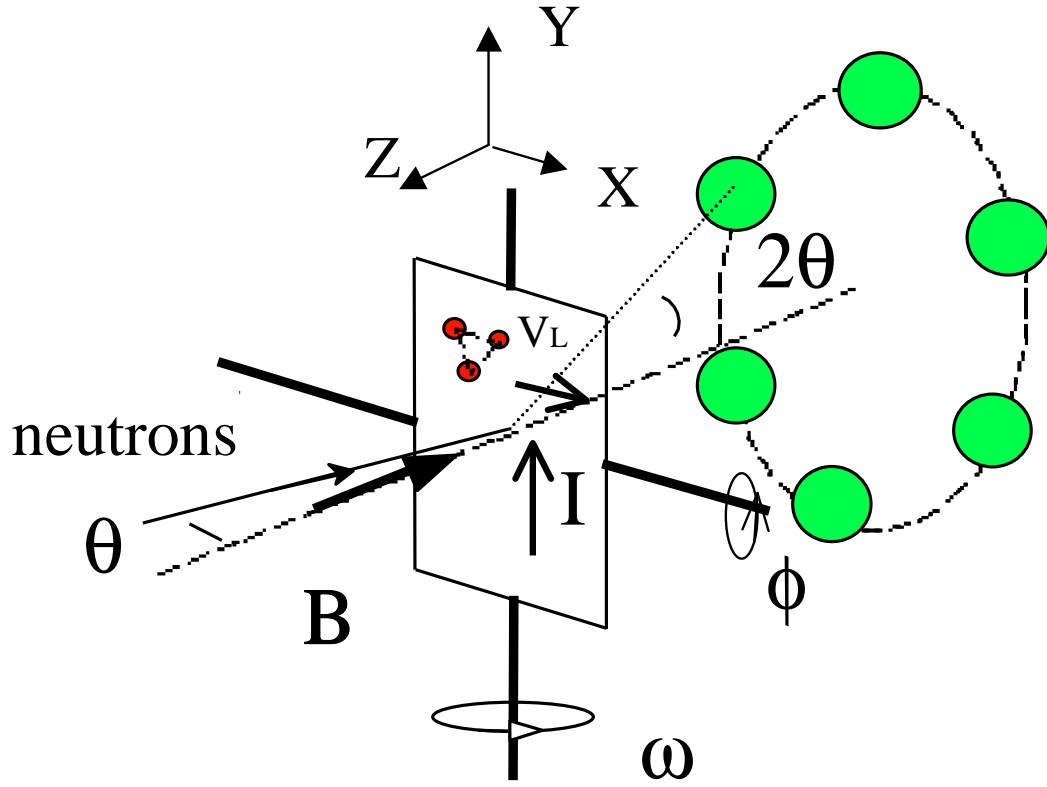


FIG. 1: The geometry of the experiment: The magnetic field  $\vec{B}$  is applied parallel to the neutron beam and is perpendicular to the large faces of the sample. The current  $I$  flows vertically and for  $I > I_c$ , vortex lines are moving perpendicular to it with a velocity  $V_L$ .

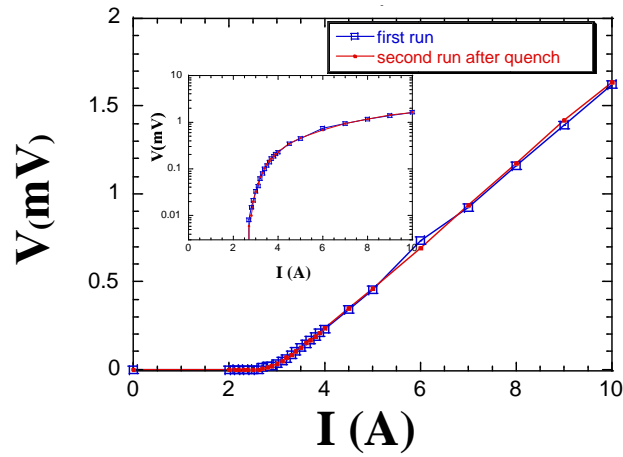
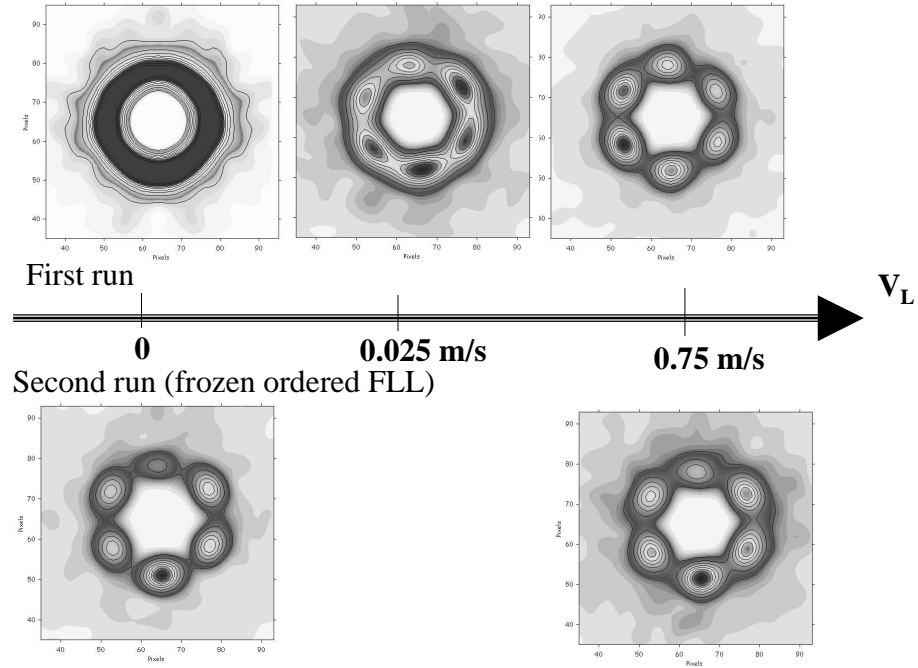


FIG. 2: a/ 2D patterns of the FLL in a polycrystalline Pb-In slab ( $T=2$  K,  $B=0.2$  T). Top: For different velocities after the equilibrium disordered state. Bottom: For different velocities after the frozen ordered state. During the record, the sample is rocked as described in the text. b/  $V(I)$  curve for the two different initial states of the FLL (square:disordered and point:ordered). In the inset is shown the same curve in a log-linear scale, so as to emphasize the perfect similarity of the curves.

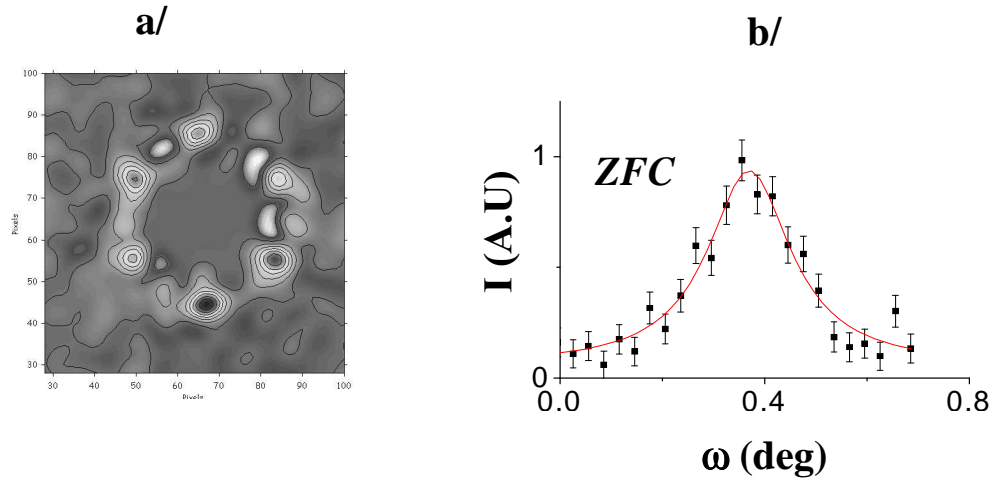


FIG. 3: a/ The diffraction pattern obtained on the multi-detector at  $2K$ ,  $0.4T$  (ZFC) for the FLL in Fe doped  $\text{NbSe}_2$ . b/ The corresponding  $\omega$ -rocking curve. The fit is a Lorentzian ( $\Delta\omega = 0.234 \pm 0.020$  deg).



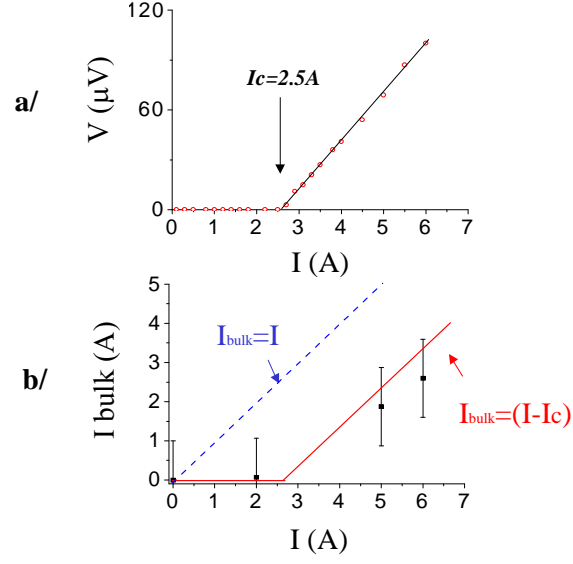


FIG. 4: a/  $V(I)$  curve after a ZFC (2K, 0.4T). One can note the linear shape  $V = R_{ff}(I - I_c)$ . b/ The Bulk current versus the transport current flowing through the sample, deduced from the broadening of the rocking curves (see text). The dotted line corresponds to a homogeneous bulk current  $I$ . The solid line corresponds to a superficial current  $I_c$  and to a bulk over critical current  $(I - I_c)$ .

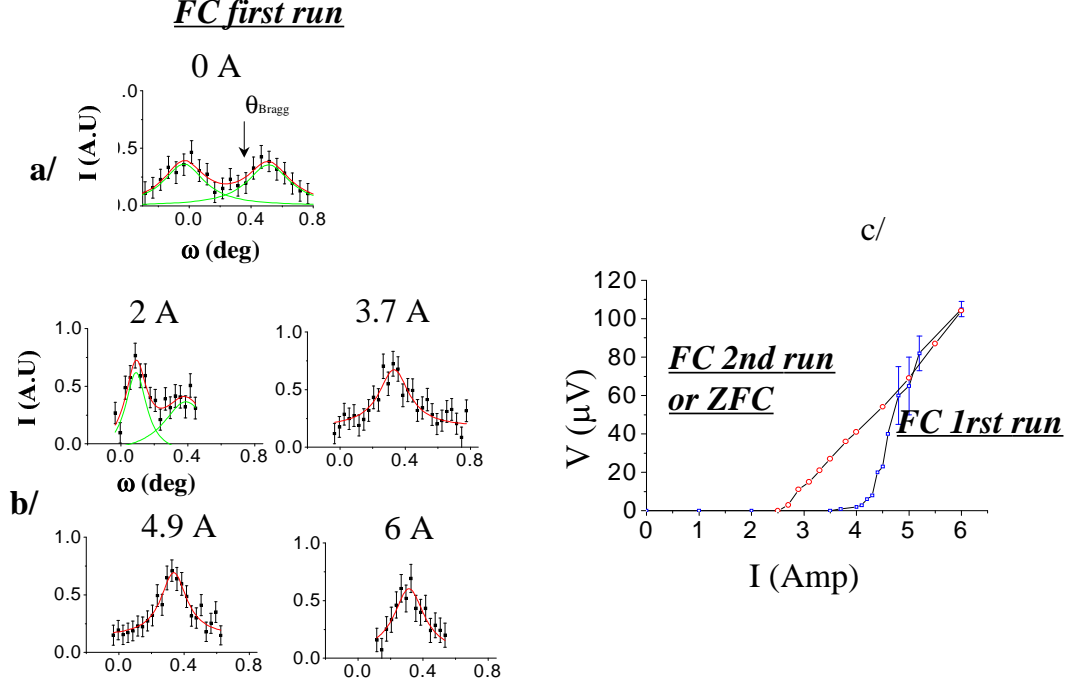


FIG. 5: a/ Rocking curve around  $\omega$  for a Bragg peak (top right) of the FC FLL. There is no applied current. The rocking curve fits with two Lorentzians. b/ The rocking curves for different values of the applied current after a FC. The high critical current is about  $4 \pm 0.5$  A. Note the disappearance of one Bragg peak for a subcritical current of 2 A. At high current, the usual shape of the rocking curve is recovered. c/ The corresponding  $V(I)$  curve after a FC (S shape), and after ZFC or for the second ramp after FC (linear shape) (2K, 0.4T).

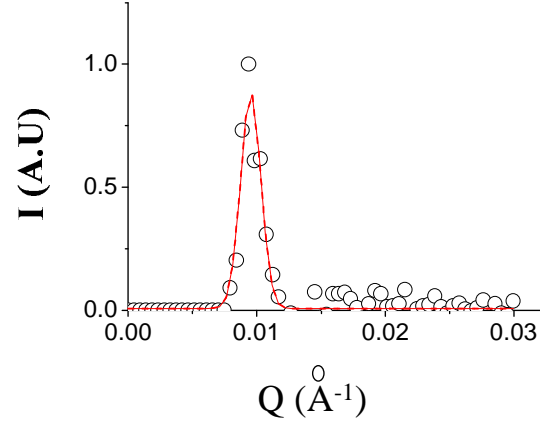


FIG. 6: The variation of the intensity diffracted from the FLL as function of the diffraction vector  $Q$  (FC,  $B=0.4\text{T}$ ). The length of the  $Q$  vector corresponds to the value fixed by the applied magnetic field of  $0.4\text{T}$ . No obvious magnetic field gradients can be observed.

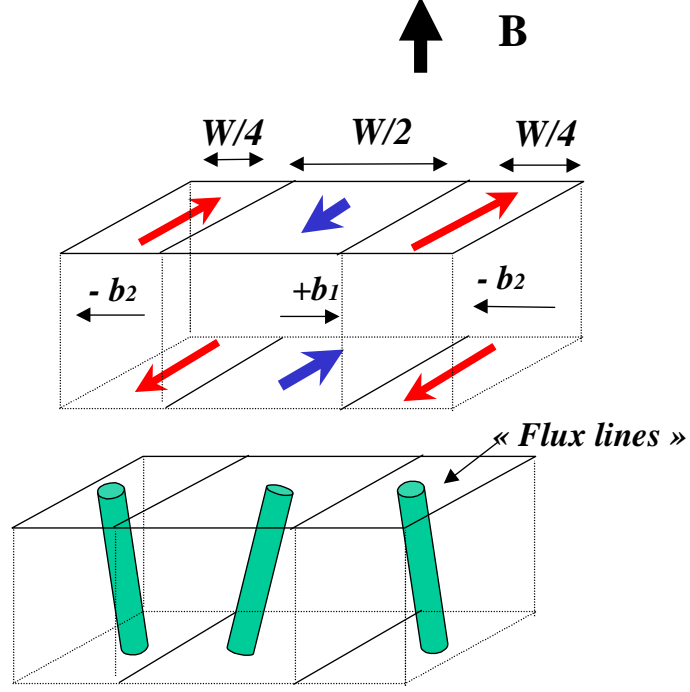


FIG. 7: One possible and highly schematic drawing of the current distribution in the sample (without applied current). Only the top and bottom surfaces have been represented. Small magnetic field components  $+b_1$  and  $-b_2$  of few Gauss are present in the plane perpendicular of the applied magnetic field. This leads to two families of tilted Flux Lines (bottom) responsible for the two Bragg peaks. For clarity, the shielding diamagnetic currents are not represented.